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(56) Documents cited
GB 2091281 A **EP 0396961 A** **US 3950259 A**

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(54) **Silica-based insulation material**

(57) A dry thermal insulation material is mouldable by compaction and comprises a mixture of finely-divided particulate silica and fibre reinforcement material, the silica comprising 50-90 wt. % of precipitated silica and the balance being pyrogenic silica.

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Thermal insulation material

This invention relates to thermal insulation material.

It is known to make microporous thermal insulation material by compacting a mixture comprising finely-divided silica powder, rutile opacifier and ceramic fibre reinforcement, as described for example in GB 1 350 661. Such material provides a very high performance thermal insulation which occupies only a limited amount of space and is relatively lightweight.

The term 'microporous' is used herein to identify porous or cellular materials in which the ultimate size of the cells or voids is less than the mean free path of an air molecule at NTP, i.e. of the order of 100 nm or smaller. A material which is microporous in this sense will exhibit very low transfer of heat by air conduction (that is collisions between air molecules). Finely-divided powders which are suitable for making such microporous materials include finely-divided aerogel. Aerogel is a gel in which the liquid phase has been replaced by a gaseous phase in such a way (e.g. drying at the critical point) as to avoid shrinkage which would occur and would contract the lattice if the gel were dried directly from a liquid. An equivalent open lattice structure can be obtained with pyrogenic (fumed) material in which the average ultimate particle size is less than 100 nm. High performance microporous thermal insulation incorporating compacted finely-divided silica powder has been available for many years, for example from Micropore International Limited under the trade mark MICROTHERM. Alumina and other metal oxides may also be used in place of silica.

Precipitation from liquid solution can also be used to prepare finely-divided powder, and if the temperature and pH are controlled during precipitation a more open lattice can be produced than would otherwise be the case. However the particles in such precipitated powders tend to clump into denser groups than is the case with aerogel or pyrogenic material, so there is greater particle-to-particle contact and inherently greater thermal conductivity. Thus, although precipitated silicas can in principle be used as thermal insulation, as suggested in GB 1 350 661, practical experience has shown that they are generally much less satisfactory than silica aerogel or pyrogenic silica. In addition it has proved necessary to compact mixtures incorporating precipitated silica to a higher density than is the case with aerogel or pyrogenic silica, in order to obtain suitable strength. Consequently the thermal insulation performance is less

satisfactory and any apparent advantage in using relatively cheap precipitated silicas in place of more expensive pyrogenic material is offset by the need for a greater thickness and higher density of insulation to obtain comparable insulation performance and strength.

Attempts have been made to develop insulation by combining the use of precipitated silica and evacuation techniques; however, these techniques are relatively expensive and require special envelopes and sealing methods. It is also very difficult to demonstrate by testing that a 'sealed' envelope is truly free of even extremely slow leaks which nonetheless would compromise the long-term integrity of the evacuated enclosure. EP 0 396 961 suggests the use of a mixture of precipitated and pyrogenic silicas, but again in the context of evacuation (to pressures of the order of 4-20 mbar) coupled with use of a gas-tight envelope.

Thus it is conventional wisdom in this art that precipitated silica is much less preferable to pyrogenic silica or silica aerogel for use in high performance thermal insulation, and indeed for many practical purposes it is unusable.

Silica aerogel and pyrogenic silica themselves have drawbacks associated with their use. Thus, in addition to relatively high cost, compacted mixtures incorporating these silicas tend to 'spring back' when the compaction pressure is removed. This springback can be as much as 20% of the size to which the mixture is initially compacted, and makes the production of moulded articles to precise dimensions very difficult and expensive. It also makes it difficult to mould such material into casings made of metal, for example, because the springback causes the moulded material to bow and bulge out of the casing. Moulding into casings typically requires use of a chemical treatment on the metal surface, as described in EP 0 246 047; however, this complicates the manufacturing process, and great care is needed in selection of a treatment agent in order to avoid damage to the structure and/or insulating properties of the insulating material.

It is an object of this invention to provide an insulating material which alleviates at least some of these problems.

According to one aspect of this invention there is provided a thermal insulation material comprising finely-divided particulate silica, the silica comprising 50-90 wt.% precipitated silica and the balance being pyrogenic silica. Preferably the silica contains 70-85 wt.% precipitated silica, and in particular 80 wt.% precipitated silica.

A mixture with these proportions of precipitated and pyrogenic silica has a thermal performance which is not too severely degraded compared to pyrogenic silica alone, and allows compacted articles of relatively low density to be produced. Furthermore, we have found that such a mixture has very low springback and can be moulded directly into metal casings without the use of a surface treatment, and will then remain securely in place without disruption of the compacted structure by internal forces in the insulation.

Typically the mixture will also contain an infra-red opacifier such as powdered rutile, and ceramic fibre to provide physical reinforcement.

An example of such a mixture has the following composition:

	% by wt
precipitated silica	48 (=80% of overall silica content)
pyrogenic silica	12 (=20% of overall silica content)
rutile opacifier	33.3
ceramic fibre	6.7

The percentages in parentheses indicate the relative proportions of the two kinds of silica.

The precipitated silica may be for example a material sold under the designation FK 320 DS by Degussa AG, Frankfurt, Germany. This material is made by precipitation followed by grinding to break up agglomerations of particles and thus reduce the apparent density; it has a BET surface area of $170\text{m}^2/\text{g}$, an average agglomerate size of $4\mu\text{m}$ and an apparent (tamped) density of $70\text{--}80\text{g/l}$. It is believed that choice of a material with a low apparent density, of this order of magnitude, is important.

The pyrogenic silica may be for example one having a BET surface area of the order of $250\text{m}^2/\text{g}$.

The constituents are mixed, for example as described in GB 1 205 572, and then the mixture is compacted in a moulding tool to the required shape and to a density of the order of 250kg/m^3 .

1. A thermal insulation material comprising finely-divided particulate silica, the silica comprising 50-90 wt.% precipitated silica and the balance being pyrogenic silica.
2. The thermal insulation material of claim 1, wherein the silica comprises 70-85 wt.% precipitated silica.
3. The thermal insulation material of claim 2, wherein the silica comprises 80 wt.% precipitated silica.
4. The thermal insulation material of any one of the preceding claims, including opacifier and/or fibre reinforcement.
5. The thermal insulation material of claim 4, comprising:
 - 48 wt.% precipitated silica;
 - 12 wt.% pyrogenic silica;
 - 33.3 wt.% rutile opacifier; and
 - 6.7 wt.% ceramic fibre.
6. The thermal insulation material of any one of the preceding claims, wherein the precipitated silica has the following characteristics:

BET surface area	170m ² /g
average agglomerate size	4µm
tamped density	80g/l.

Amendments to the claims have been filed as follows

1. A dry thermal insulation material mouldable by compaction to a required shape, said material comprising a mixture of finely-divided particulate silica and fibre reinforcement material, the silica comprising 50-90 wt.% precipitated silica and the balance being pyrogenic silica.
2. The thermal insulation material of claim 1, wherein the silica comprises 70-85 wt.% precipitated silica.
3. The thermal insulation material of claim 2, wherein the silica comprises 80 wt.% precipitated silica.
4. The thermal insulation material of any one of the preceding claims, wherein the fibre reinforcement material comprises ceramic fibre.
5. The thermal insulation material of any one of the preceding claims, including an opacifier.
6. The thermal insulation material of claim 5, comprising:
48 wt.% precipitated silica;
12 wt.% pyrogenic silica;
33.3 wt.% rutile opacifier; and
6.7 wt.% ceramic fibre
7. The thermal insulation material of any one of the preceding claims, wherein the precipitated silica has the following characteristics:

BET surface area	170m ² /g
average agglomerate size	4µm
tamped density	80g/l.
8. A moulded shape formed by compaction of the thermal insulation material according to any one of the preceding claims.
9. A moulded shape according to claim 8, having a density of the order of 250kg/m³.

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Patents Act 1977
Examiner's report to the Comptroller under
Section 17 (The Search Report)

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Relevant Technical fields

(i) UK CI (Edition K) C1H, C1J

(ii) Int CI (Edition 5) C04B

Search Examiner

MISS M M KELMAN

Databases (see over)

(i) UK Patent Office

(ii)
Online Databases: WPI, Chemeng

Date of Search

14 AUGUST 1991

Documents considered relevant following a search in respect of claims

1 to 6

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
X	GB 2091281 A (RHONE-POULENC) see the Examples	1,2,3,6
X	EP 0396961 A1 (DEGUSSA) see page 3 lines 22 to 24 and 39 to 40	1,4 at least
X	US 3950259 A (JOHNS-MANVILLE) see Claim 10, column 4 lines 9 to 29 and the Examples	1,4 at least

SF2(p)

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Category	Identity of document and relevant passages	Relevant to claim(s)

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family, corresponding document.

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